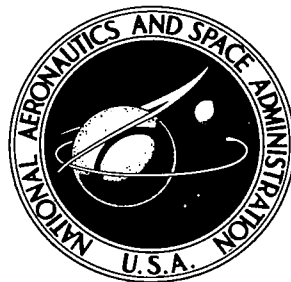


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# THE INFLUENCE OF GASEOUS ENVIRONMENT ON THE SELF-ADHESION OF METALS

*by William P. Gilbreath*

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## TABLE OF CONTENTS

	<u>Page</u>
SUMMARY . . . . .	1
INTRODUCTION . . . . .	1
SYMBOLS . . . . .	2
EXPERIMENTAL PROCEDURE . . . . .	3
ADSORPTION RATE THEORY . . . . .	4
RESULTS AND DISCUSSION . . . . .	7
Mechanical Testing and Impurity Effects . . . . .	7
Gaseous Environment Effects . . . . .	9
CONCLUDING REMARKS . . . . .	16
REFERENCES . . . . .	18
TABLES . . . . .	21

# THE INFLUENCE OF GASEOUS ENVIRONMENT ON THE SELF-ADHESION OF METALS

By William P. Gilbreath

Ames Research Center

## SUMMARY

The self-adhesion of metal surfaces was measured in various gases with pressure, exposure duration, and temperature as variables. Aluminum, copper, gold, lead, magnesium, and titanium were studied in air, argon, carbon monoxide, ethylene, hydrogen, nitrogen, and oxygen.

The results showed that loss of adhesion for a particular metal resulted from gas adsorption on its contacting surface and that the effect of environment upon adhesion could be related to the heat of adsorption for the particular metal-gas interaction.

## INTRODUCTION

Adhesion, or cold-welding, is known to be an environment-sensitive property of metals. Contaminating surface layers, commonly oxides, water, or oil, inhibit the adhesion of most metals. Numerous investigators (e.g., refs. 1-4) have shown that small amounts of oxygen or air significantly decrease the adhesion of initially clean surfaces. Although the effect of various gaseous environments upon wear (ref. 5) and friction (refs. 2,3,6,7,8) has been studied, comparable work has not been published in the related field of adhesion. In the present study, the influence of various gaseous environments on the self-adhesion of a number of metals with initially clean surfaces was investigated and the observed effects have been related to chemical or physical properties of the interacting gas metal species.

The purpose of this study was to investigate the relationship between adhesion and gas environment and thereby to help develop guidelines useful in the selection of metals for a specific operation in particular gas environments. For example, metal-environment combinations found to cause relatively high adhesion would be expected to result in more unwanted wear, and higher friction and contact seizure than combinations producing little or no adhesion.

## SYMBOLS

k	Boltzmann constant, $1.38 \times 10^{-16}$ erg ( $^{\circ}\text{K}$ ) $^{-1}$
M	molecular mass of molecule, g-mole $^{-1}$
m	mass of molecule, g-mole $^{-1}$
n	molecular density, molecules cm $^{-3}$ or molecules cm $^{-2}$
P	pressure, torr
R	gas constant, 2 g-cal ( $^{\circ}\text{K}$ ) $^{-1}$ mole $^{-1}$
s	sticking coefficient
T	temperature, $^{\circ}\text{K}$
t	time, sec
$\alpha$	adhesion coefficient
$\bar{\alpha}$	normalized adhesion coefficient
$\Delta H$	heat of adsorption, kg cal mole $^{-1}$
$\nu$	molecular flux, molecules cm $^{-2}$ sec $^{-1}$
$\sigma$	adsorbed molecular surface density, molecules cm $^{-2}$
$\tau$	mean surface residence time of adsorbed molecules, sec
$\tau_0$	oscillation period of adsorbent surface atoms, sec

### Subscripts

a	adsorbed
d	desorbed
e	equilibrium
g	gas
i	value of ith test
s	solid

## EXPERIMENTAL PROCEDURE

Self-adhesion values for the metals tested were found by measuring the tensile force necessary to separate two surfaces previously pressed together with a given compressive load under the controlled conditions described below. These values were expressed as an adhesion coefficient,  $\alpha$ , the ratio of the measured tensile force (adhesion) to the preimposed compressive joining force. For this study, initially clean surfaces were produced by fracturing notched cylindrical specimens with a tensile load. In order to retain these clean surfaces for a reasonable length of time (necessary to permit study of the effect of various gas environments on them), the initial fracturing was done in ultra-high vacuum. The apparatus (fig. 1) consisted of a vacuum chamber,

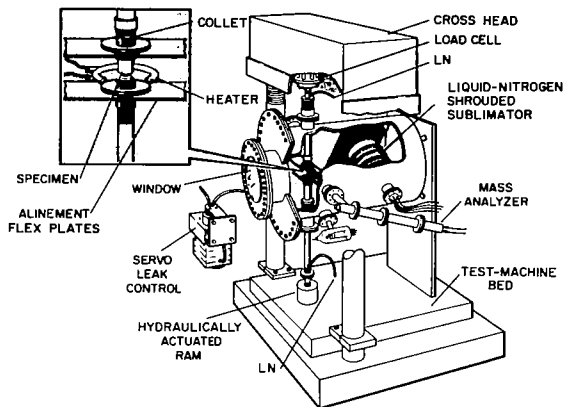


Figure 1.- Test apparatus used to investigate the influence of environment on adhesion.

a hydraulic test machine for initially fracturing the specimen and for applying the compressive and tensile loads during adhesion tests, a gas manifold and servo-controlled leak for establishing various environments, a system for heating and cooling the specimen, gages for analyzing the environment, and related instrumentation.

The vacuum chamber was a stainless steel belljar, 28 cm in diameter by 38 cm long. It was evacuated to pressures below  $1 \times 10^{-10}$  by a trapped mechanical pump, ion pumps, and a liquid-nitrogen-shrouded titanium sublimation pump. The metal specimens were screwed into oxygen free, high conductivity (OFHC) copper collets, which

were, in turn, attached to bellows-sealed, double, extra heavy-walled tubing that served as drive rods. The lower rod was connected to the hydraulically actuated ram of the test machine and the upper rod was connected to a load cell. Flex plates, 0.076 cm thick, aligned the specimen halves for rejoining after fracturing but did not contribute to the load cell output as both plates were anchored to the chamber at their ends and the upper plate was immobile in relation to the load cell.

After the specimen was initially fractured to expose a clean surface, the closed-loop, servo-controlled test machine was set to follow a program in which a number of accurately reproducible mechanical test cycles could be run automatically. A single mechanical test cycle for all metals except lead consisted in rapidly joining the fractured halves and holding them under  $45 \pm 0.2$  kg load for 3.0 seconds, rapidly unloading, fracturing at a ram speed of 0.007 cm/sec, rapidly separating them to about 1 cm, and then exposing them to the environment for about 30 seconds before repeating the cycle. For lead, it was necessary to use a 22-kg load and a ram speed of 0.05 cm/sec because of its small compressive yield strength and large creep rate. Adhesion (the fracturing load) of less than 40 g could be detected. This corresponded to an adhesion coefficient,  $\alpha$ , of less than 0.001 (0.002 for lead). The compressive loads used were of such magnitude as to cause from 2 to 15 percent (depending

upon the metal and its compressive yield strength) of the fractured cross section to be in actual contact during the weld part of the test cycle. For experiments using impulsive loading, the specimens were loaded at a frequency of 15 Hz for several minutes at an amplitude of 0 to 45 kg.

System pressures were measured by nude and tubulated hot cathode gages and thermocouple gages in the applicable pressure ranges. The residual gas environment and the artificially created gas environments were measured by means of a quadrupole mass analyzer with electron multiplier at pressures below  $1 \times 10^{-4}$  torr. The desired gaseous environment was admitted to the system through transfer regulators, carefully purged lines, and a servo-controlled leak valve which maintained a preselected pressure. The usual practice was to evacuate the system to about  $1 \times 10^{-10}$  torr and then continually admit the gas at the desired level of pressure while keeping the ion pumps running. This dynamic procedure in most instances maintained the absolute pressure of the background gases at about  $10^{-10}$  torr; thus, the purity level of the environment was essentially that of the admitted gas. When pressures above  $1 \times 10^{-5}$  torr were to be used, the pumps were shut off, since at these pressures, the pumps heated and regurgitated impure gases. Under these static conditions of pumps off and no gas flow, somewhat larger absolute impurity levels of background gases are to be expected. At pressures greater than  $10^{-4}$  torr no check on the environment was made since the analyzer could not be operated in this range.

In the range from  $-150^{\circ}$  to  $+500^{\circ}$  C, the specimen was cooled by liquid nitrogen circulating to the collets through the tubular drive rods and heated by a 2000-W quartz lamp that encircled the specimen. Thermocouples attached to the upper and lower portions of the sample indicated that temperatures were controlled to within  $5^{\circ}$  C in the above range.

Two specimen configurations were used: in most experiments, the specimens were 1.27-cm-diameter rods, 2 to 5 cm long, threaded on both ends, and machined with a  $60^{\circ}$  notch angle to 0.636 cm diameter. For some experiments gold disks, 0.3 cm thick by 0.6 cm diameter were silver-soldered to threaded copper rods 1.27 cm in diameter which were in turn inserted into the collets. Specimen materials and their purities were: aluminum (99.99 percent), copper (OFHC), gold (99.999 percent), lead (99.99 percent), magnesium (99.95 percent), and titanium (99+ percent).

Gases, used for creating the artificial test environments, and their minimum purities were: air (laboratory), air (dry, hydrocarbon free), argon (99.999 percent), carbon monoxide (99.5 percent), ethylene (99+ percent), hydrogen (99.999 percent), nitrogen (99.99 percent), and oxygen (99.99 percent).

#### ADSORPTION RATE THEORY

If, in an adhesion test, a variation in adhesion can be attributed to the gaseous environment, the variation must be due to adsorption of the gas on the initially clean metal surface. The presence of an adsorbed species will reduce, and possibly entirely eliminate, the metal-metal atomic bond formation expected to occur on contact of the metal surfaces. The number of bonds



formed on contact, and thus the measured adhesion, must, in some way, be related to the concentration of adsorbed species on the surface, which in turn depends upon several well-established parameters. These parameters are (1) the number of gas molecules impinging on the surface (pressure), (2) the ratio of those molecules sticking on the surface to the number impinging (sticking coefficient), and (3) the mean lifetime of the adsorbed species on the surface.

For less than monolayer adsorption, an approximation of the surface coverage,  $\sigma$ , in molecules per  $\text{cm}^2$ , after exposure for a time,  $t$ , in seconds to a gas at a given pressure,  $P$ , in torr will now be derived.

The rate of change of surface coverage is given by the difference of adsorption and desorption rates of  $n$  molecules or

$$\frac{d\sigma}{dt} = \frac{dn_a}{dt} - \frac{dn_d}{dt} \quad (1)$$

The rate of adsorption depends upon the sticking coefficient,  $s$ , and the particle flux,  $\nu$ , as

$$\frac{dn_a}{dt} = \nu s = P(2\pi mkT)^{-1/2} s = 3.5 \times 10^{22} s P (MT_g)^{-1/2} \text{ sec}^{-1} \text{ cm}^{-2} \quad (2)$$

The right hand equivalent follows from gas kinetic theory;  $m$ ,  $M$ ,  $k$ , and  $T_g$  are the mass of molecule, molecular weight (g/mole), Boltzmann constant, and temperature ( $^{\circ}\text{K}$ ) of the gas, respectively. The desorption rate is proportional to the number of adsorbed molecules and inversely proportional to their mean residence time,  $\tau$ ,

$$\frac{dn_d}{dt} = \frac{\sigma}{\tau} \quad (3)$$

Substituting equations (2) and (3) in (1) yields

$$\frac{d\sigma}{dt} = s\nu - \frac{\sigma}{\tau}$$

which, after integration becomes

$$\sigma = s\nu\tau(1 - e^{-t/\tau}) \text{ molecules/cm}^2 \quad (4)$$

Frenkel (ref. 9) showed that the mean resident time,  $\tau$ , could be related to the heat of adsorption of the gas on the surface at temperature  $T_s$ , by

$$\tau = \tau_0 e^{\frac{\Delta H}{RT_s}} \quad (5)$$

in which  $\tau_0$  is a measure of the oscillation period of the surface atoms of the adsorbent and is normally from  $10^{-12}$  to  $10^{-14}$  second. Equation (4) provides an indication of the degree of surface coverage under a set of specific test conditions. For a gas exhibiting short  $\tau$  (and/or under conditions of long  $t$ ), equation (4) reduces to the equilibrium form,  $\sigma_e$ , given by

$$\sigma_e = s\sqrt{\tau} \quad \text{or from (2)} \quad (6)$$

$$\sigma_e = 3.5 \times 10^{22} \text{ sPr}(\text{MT}_g)^{-1/2} \text{ molecules/cm}^2 \quad (6a)$$

Conversely, for long  $\tau$  (or short  $t$ ) and low fluxes, the pre-equilibrium condition is important and equation (4) can be initially approximated (from the series expansion of the exponential):

$$\sigma = s\sqrt{t} \quad (7)$$

$$\sigma = 3.5 \times 10^{22} \text{ sPt}(\text{MT}_g)^{-1/2} \text{ molecules/cm}^2 \quad (7a)$$

Thus, depending primarily upon the magnitude of  $\tau$ , and hence of  $\Delta H$ , equations (6) and (7) and, therefore, pressure and integrated exposure ( $\int_0^t P \, dt$ ), will have varying degrees of importance in dictating adsorbed gas coverage. For example, for typical chemisorbable gases,  $\tau$  is long (e.g., about  $10^{17}$  seconds for a  $\Delta H$  of 40 kcal/mole), so that the surface coverage will increase as a function of the product of  $P$  and  $t$  (i.e., integrated exposure) as indicated in equation (7). On the other hand, for a typical physisorbable gas,  $\tau$  is short (e.g., about  $10^{-10}$  sec for  $\Delta H$  of 4 kcal/mole) and the equilibrium condition as given by equation (6) is virtually instantaneously attained, in which case the significant experimental variable is the pressure.

In adhesion experiments in chemisorbable gases, the environmental effect was found to be cumulative. That is, a metal specimen, after initial fracture, exposed to gas at pressure  $P_1$  for a time  $t_1$ , joined, refractured, and exposed again for  $t_2$  at  $P_2$ , or for  $n$  test cycles  $t_n$  at  $P_n$  would have a cumulative exposure equal to  $\sum_{i=1}^n (Pt)_i$ , or more precisely,  $\int_0^t P \, dt$  (integrated exposure), allowing for variation in  $P$  during a single exposure. The effect on adhesion of a given total integrated exposure, occurring over  $n$  weld-break-exposure cycles, was found (as shown later) to be similar to the effect of a single equivalent integrated exposure. Thus, it was possible to plot the results as a function of the effect of integrated exposure on some adhesion parameter. The parameter can be simply the adhesion coefficient,  $\alpha$ , as previously defined. However, to get a true picture of the gas environment effect on metals (without the overlapping effects of mechanical testing) a normalized adhesion coefficient,  $\bar{\alpha}$ , is more appropriate, it is derived by relating the result in the gas environment to that in ultra-high vacuum (UHV). Specifically, the adhesion coefficient,  $\alpha_i$ , obtained after a given number of weld-break cycles in the gas environment is multiplied by the ratio of  $\alpha$  of the test after the first cycle,  $\alpha_1$ , to that after the given number of cycles,  $\alpha_i$  in UHV; that is,

$$\bar{\alpha} = \frac{[\alpha_1]_{\text{gas}}}{[\alpha_1]_{\text{UHV}}} [\alpha_1]_{\text{UHV}} \quad (8)$$

## RESULTS AND DISCUSSION

The first part of this section will be devoted to discussing two phenomena, mechanical testing and impurity effects, which tend to obscure the expected gas-environment effects when the test technique of this study is employed. In the last and major part of this section, data will be presented on the effects of a number of gaseous environments on the self-adhesion of the metals studied. These latter results will be interpreted primarily in relationship to the heat of adsorption for a given combination of gas environment and metal.

### Mechanical Testing and Impurity Effects

With many of the metals examined, the change in adhesion caused by the gas environment was unfortunately similar to the effect produced by repetition of the weld-break, mechanical test cycle itself. In order to isolate and assess the influence of this latter factor on the results, the test cycle was repeated in an ultra-high vacuum in the case of every metal studied. At such vacuums ( $10^{-9}$  to  $10^{-11}$  torr), the gas concentration is so low that, within the test period, its influence on the initially clean surfaces and, thus, on adhesion may be neglected. (Mass spectrometric gas analysis of this vacuum environment showed the residual gases to be:  $H_2$ , CO,  $CH_4$ ,  $CO_2$ ,  $H_2O$ ,  $N_2$ , and  $O_2$ .) An example of the effect of the weld-break test cycle on the adhesion of aluminum is shown in figure 2. The results of three ultra-high

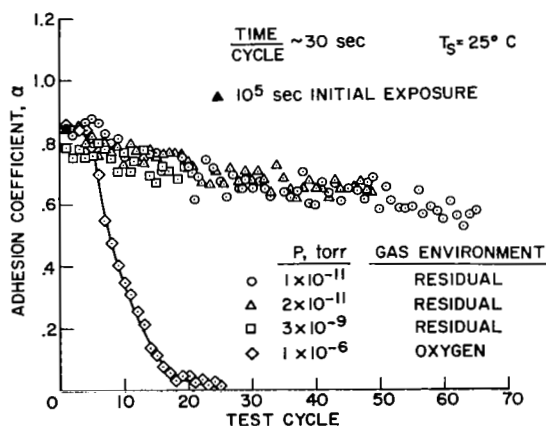


Figure 2.- Comparison of adhesion characteristics of 99.99 percent aluminum in UHV and oxygen.

vacuum experiments, each performed with about a 30-second interval between individual weld-break cycles, and, in one case,  $10^5$  seconds exposure before the first test, are independent of exposure and pressure and appear to depend only on the number of prior test cycles. Likely explanations for the noted decrease of adhesion with repeated mechanical testing are surface effects due to (1) a change in mechanical properties (e.g., hardness) of the metal, (2) a variation in topography with testing, and/or (3) a worsening lattice match (refs. 1, 10).

of initially clean aluminum surfaces. The major distinction between the weld-break effect and that engendered by the gas environment is that, in the latter case, the effect on  $\alpha$  is large. From the example shown in combination with other data to be presented later, it was concluded that the environmental effect on adhesion is generally independent of the number of test cycles and may be many times larger per test cycle than the weld-break effect. On this

On the same figure, for comparison, data are also presented from a typical run showing the effect of a chemisorbable gas ( $O_2$ ) on the adhesion behavior

basis, the simple normalizing technique described earlier (eq. (8)) was developed to exclude the effects of mechanical testing from this study of the effects of gas environment on adhesion. Data showing the effect of mechanical testing in UHV upon the adhesion coefficients of metals examined in the present study are presented in table I.

It is also important to consider the effect of impurities at the surface when trying to assess the influence of a gas environment on adhesion. Interfering impurities have three sources: the specimen bulk, the neighboring specimen surface, and the test gas environment. Diffusion of impurities from the bulk to the initially clean surface may not be important at the temperatures and times used in the experiments (ref. 11). Surface diffusion, which has an activation energy that is lower than bulk diffusion and typically according to (ref. 11) one-third that of desorption (itself a significant phenomenon, eq. (5)) might be considered a more likely interfering process. However, it appears (refs. 11, 12) that, from a practical standpoint, surface diffusion also would have little effect on adhesion under the test conditions used herein. That is, the total flux of low mobility contaminants (such as oxides) onto the clean surface from adjoining surfaces would be small because of the relatively short time for a given experiment, while highly mobile species would be desorbed before moving appreciable distances on the surfaces. Substances of moderate, as well as high, mobility would have been desorbed during the bakeout and pumpdown prior to each experiment. The assumptions that both surface and bulk diffusion were low was verified, at least for aluminum, in the one experiment in figure 2 in which the specimen, after initial fracturing, was exposed for  $10^5$  seconds at  $10^{-11}$  torr before the initial weld-break test. Since subsequent mechanical testing produced results similar to the other ultra-high vacuum tests in figure 2, it was concluded that surface diffusion, and also bulk diffusion, of contaminants to the clean surface did not occur to a measurable degree. Interfering impurities from the third source, the test gas itself, can definitely affect the results. The apparent effects of these gaseous impurities on the results are discussed below.

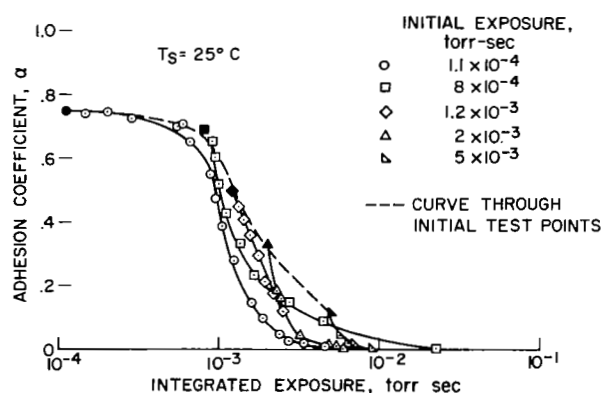


Figure 3.- Effect of oxygen on the adhesion coefficient of OFHC copper after various initial exposures.

Figure 3 presents results showing the adhesion behavior of copper in oxygen environments obtained after five different exposure intervals after the initial fracturing but prior to the initial weld-break tests. The initial points on each curve (filled symbols) reflect only the effect of contamination with oxygen and none of the complexities, as described earlier, which arise through repeated mechanical testing. These initial points fall on a smooth curve (dashed line). Although they do not deviate much from the remainder of the data points, which were determined from subsequent repeated

mechanical testing at the test conditions noted, their location relative to

the other data confirms that the effect of repeated testing is to reduce adhesion somewhat more rapidly than it would be reduced by the environment alone.

### Gaseous Environment Effects

Aluminum.— The adhesion behavior of aluminum in various gas environments serves as a good illustration since (1) aluminum reacted in two distinct ways, which apparently depended on its interaction with the gaseous environment, (2) the effects of the environment were clearly distinguishable from other factors affecting the results, and (3) the interaction of clean aluminum surfaces with most of the gases used was fairly well defined. The results for aluminum will be discussed in detail and, since the results for the other metals tested are similar, the behavior of these metals can, in most instances, be referenced to the conclusions found from examining the aluminum system.

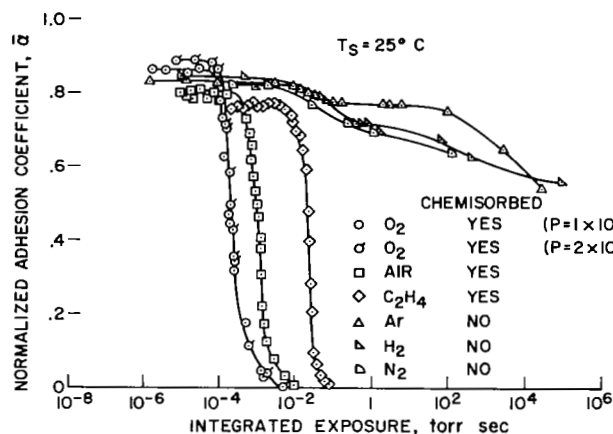


Figure 4.— Effect of various gas environments on the normalized adhesion coefficient of 99.99 percent aluminum.

Figure 4, showing how the normalized adhesion coefficient for aluminum is affected by exposure to various gases, clearly distinguishes the two types of interaction. Gases that are chemically adsorbable (arbitrarily defined as heats of adsorption above 20 kcal/mole, ref. 13) such as oxygen, air, or ethylene, have a greater and quicker effect on measured adhesion than those that are physically adsorbable (heats of adsorption below 5 kcal/mole, ref. 13), such as argon, nitrogen, or hydrogen. In the former group of gases, a precipitous decrease in adhesion is seen after an exposure of  $10^{-4}$  to  $10^{-2}$  torr sec. In oxygen, this exposure amounted to about

$1 \times 10^{-4}$  torr sec. In considering the degree of surface contamination to which this exposure corresponds, we see that  $1 \times 10^{-4}$  torr sec is considerably greater than the often-stated (e.g., ref. 14) exposure of  $2 \times 10^{-6}$  torr sec for the formation of a "theoretical" monolayer. However, on the basis of more realistic approximations, it is not much greater than the actual exposure required for monolayer formation. These approximations are derived from (a) a modification of gas impingement rate on a given specimen surface based on the eclipsing of molecular flow by the presence of the other sample half (see fig. 1), (b) evaluation of the surface area or roughness factor of the fractured specimen which corrects for the difference between true surface area and that surface area based on cross-sectional dimensions, and (c) consideration of the fraction of these impinging molecules that rebound from the surface rather than stick. Accordingly, the "theoretical" monolayer formation time would be multiplied by the following approximate factors: (a) by 2, based on calculations of the gas impingement rate in experimental configuration used, (b) by about 8, based on consideration of measured gross surface roughness and cited (ref. 15) specific-surface measurements, and (c)

by roughly 3, based on typical sticking coefficients (ref. 16). Thus, actual monolayer coverage might not occur until an exposure of about  $10^{-4}$  torr sec ( $2 \times 10^{-6}$  torr sec  $\times 2 \times 8 \times 3$ ) is reached. From this it can be seen that coverage, sufficient to cause the complete loss of adhesion noted in figure 3, is probably no more than several monolayers. It is of interest to note from work on fatigue of metals in vacuum and air (refs. 17-20) that changes in fatigue life also occur at exposures of  $10^{-2}$  to  $10^{-4}$  torr sec. From the data in figure 4 for aluminum in air, it is seen that approximately a fivefold increase in exposure was necessary to cause a change in  $\alpha$  comparable to that found in  $O_2$ , suggesting that  $O_2$  is the active gas in air (20 percent  $O_2$  in air). For those tests conducted in ethylene ( $C_2H_4$ ), a similar precipitous decrease in  $\alpha$  occurred, but at greater exposures, which agrees qualitatively with the fact that  $C_2H_4$  with its lower heat of adsorption is more slowly adsorbed than  $O_2$  (refs. 13, 16).

In general, it was found (as predicted by eq. (7)) that in the case of gases with high heats of adsorption, the shapes and positions of the  $\bar{\alpha}$ -integrated exposure curves were independent of pressure. This is illustrated in figure 4 in which the results for experiments performed in oxygen, one at a constant pressure of  $2 \times 10^{-6}$  torr and the other at a pressure of  $1 \times 10^{-7}$  torr, are virtually coincidental.

One particular type of experiment, conducted a number of times with aluminum and other materials in various environments, showed clearly that contamination of the surface from the environment was causing the loss of adhesion. In these tests, after an active gas had caused a partial loss of adhesion, the active gas (usually  $O_2$ ) was pumped out until the chamber pressure was less than  $1 \times 10^{-9}$  torr. Adhesion testing was then continued but no further decrease in  $\alpha$  occurred; in fact, it was more usual to observe that the adhesion increased somewhat. In the case of aluminum in  $O_2$  and most other metal-gas combinations in which the interacting gas was strongly adsorbed, this increase amounted to a few percent and was possibly due to the "plowing-up" of new clean surface from the bulk during compressive loading. If the active gas, at the same pressure, were then readmitted, the adhesion would again drop sharply.

Decreases in the adhesion coefficient also occurred in the environments which contained, supposedly, only gases ( $H_2$ ,  $N_2$ , and Ar), which were weakly adsorbable by aluminum (fig. 4). However, the losses in these gaseous environments occurred at greater exposures than in air,  $O_2$ , and  $C_2H_4$ ; it was not as precipitous, and usually total loss of adhesion did not occur. From equation (6), which applies to metal-gas systems of low  $\Delta H$ , surface coverage in such systems is not dependent on integrated exposure; rather it is dependent on pressure and mean residence time. However, the data were still plotted on the same coordinate system of figure 4 to emphasize the large magnitude of behavioral difference between aluminum in chemisorbed and physisorbed gases. Furthermore, in the experimental procedure used, results for the two classes of gaseous environments may be directly compared on a single plot. To deal with convenient run times, the pressures used were, in most cases, roughly  $10^3$  less than the corresponding integrated exposures shown, thus  $P$  generally increased with increasing  $P \times t$ . There are two possible explanations for the partial adhesion loss at higher exposures: (1) Some quantity of the inert, physisorbable gas sufficient to affect adhesion is adsorbed at higher

pressures, or (2) active impurities in the otherwise inert gas are being adsorbed and affect adhesion when the product of their partial pressure and exposure time reaches about  $10^{-3}$  torr sec.

Explanation (1) will be discussed with reference to equations (5) and (6), which indicate the degree of adsorption expected under equilibrium conditions for various test conditions. For example, for nitrogen on aluminum at room temperature,  $\tau$  would have a value of  $10^{-10}$  second (assuming purely physical adsorption and a  $\Delta H$  of 4 kcal/mole (ref. 12)). According to equation (6a), monolayer coverage (about  $5 \times 10^{14}$  atoms/cm<sup>2</sup>) would not occur below a nitrogen pressure of several atmospheres. Based on the maximum pressures of a few torr used to generate the data in figure 4, the expected surface concentrations of adsorbed gases should be less than 1 percent and, thus, should be too low to greatly affect adhesion. (However, the possible occurrence of higher energy sites under these pressures might lead to stronger adsorption which, in turn, would cause loss of adhesion.)

Explanation (2) is certainly plausible since hydrocarbon, water, and oxygen impurities, in the gases used, amounted to several parts per million. As an example, a concentration of 1 part of oxygen in  $10^8$  parts of argon could cause the reduction in  $\bar{\alpha}$  noted at an exposure of  $\sim 10^5$  torr sec. Impurities probably played a role in some earlier experiments (not shown) with aluminum in industrial grade hydrogen (99.8 percent, assay) in which  $\bar{\alpha}$  dropped below 0.1 at less than 1 torr sec exposure. It is difficult either to measure or estimate the impurity concentration in the gaseous environment since an unknown amount could be inadvertently added in the gas transfer process. (As indicated earlier, the partial pressure analyzer could not be used at the pressures pertinent to this part of the study.) This explanation would also account for the poor reproducibility in some tests.

It is assumed that both factors discussed above probably contribute to some extent to the loss of adhesion observed when aluminum was tested in physisorbable gases. It is likely that, when the loss in  $\bar{\alpha}$  is relatively large, the principal factor causing the loss is the adsorption of active impurities from the test gas environment.

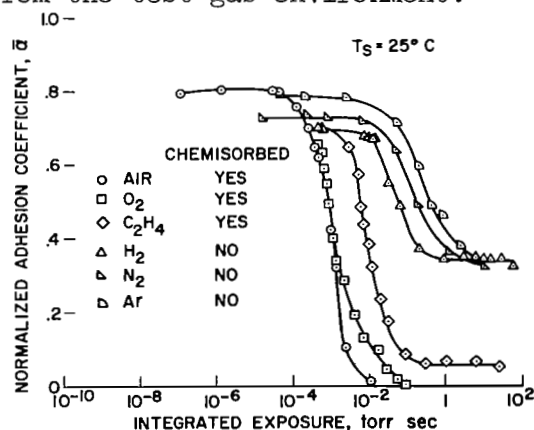


Figure 5.- Effect of various gas environments on the normalized adhesion coefficient of OFHC copper.

Copper.- The effect of various gaseous environments on the adhesion of copper was studied, and typical results are shown in figure 5. The data are similar to those for aluminum (fig. 4) in that chemisorbable gases had a much greater influence on adhesion than physisorbable ones did. Thus, the environments with a relatively large  $\Delta H$  ( $O_2$ , air, and  $C_2H_4$ ) caused complete or nearly complete loss of adhesion at exposures of  $10^{-3}$  to  $10^{-1}$  torr sec while those environments with a relatively small  $\Delta H$  (Ar,  $N_2$ , and  $H_2$ ) caused a decrease in  $\bar{\alpha}$  to about 0.3 at exposures of about  $10^{-1}$  torr sec.

These results are in accord with Trapnell's observation (ref. 13) that aluminum and copper exhibited similar adsorptive behavior toward various gases. The partial loss of adhesion in  $N_2$ ,  $H_2$ , and Ar environments occurred at pressure levels much too low to be explained by equations (5) and (6) above. Perhaps preferential impurity adsorption could explain this. The  $\bar{\alpha}$  values of 0.3 to 0.4 are stable and are relatively independent of further exposure. For example, an  $\bar{\alpha}$  of 0.4 could be maintained in 1 torr  $H_2$  indefinitely ( $\sim 10^5$  sec). Unfortunately, because of bursting strength limitations of the test chamber used and because of the increase in the partial pressure of active impurities, with the test technique employed, the complete loss of adhesion expected (eq. (6)) to occur in these weakly adsorbed gases ( $\Delta H \sim 5$  kcal/mole) at pressures above one atmosphere could not be unambiguously demonstrated.

According to equation (5), varying the surface temperature will change the mean residence time of a gas on a surface, and thus also surface coverage (eq. (6)). By this means, a metal at a given pressure-exposure condition might show no effect of environment on its adhesion at one temperature and yet might show a strong adhesion dependency toward the gas environment at lower temperature. It was possible by lowering the temperature to demonstrate the same effect on adhesion, of the gas environment, as increased pressure would show, as will be discussed next.

The adhesion of copper in carbon monoxide was studied using pressure, exposure, and surface temperature as the test parameters. The adhesion of copper in this environment should reflect the intermediate (10 to 20 kcal/mole) adsorption character of this system. That is, at temperatures below a certain level and/or pressure above a certain level, adsorption sufficient to cause significant loss of adhesion should occur. The results of a study that demonstrate this effect are presented in figures 6 and 7 and table II.

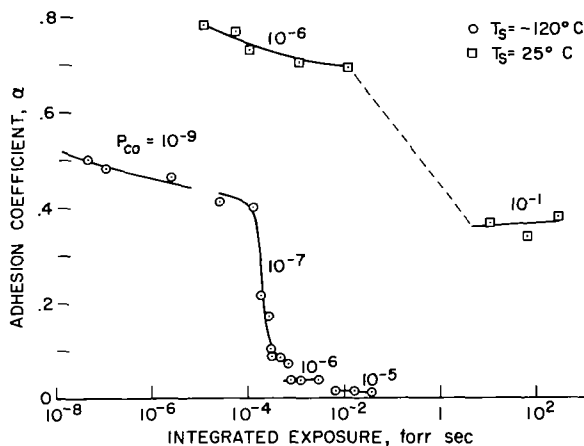


Figure 6.- Effect of exposure to carbon monoxide on the adhesion coefficient of OFHC copper at two temperatures.

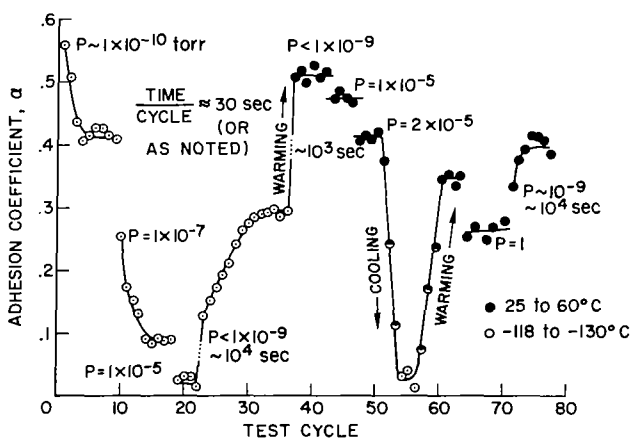


Figure 7.- Effect of temperature and pressure of carbon monoxide on the adhesion coefficient of OFHC copper.

Figure 6 shows the adhesion behavior of copper in carbon monoxide at two temperatures and several pressures as a function of exposure. At  $25^\circ\text{C}$  and  $10^{-6}$  torr, carbon monoxide is shown to have little effect on adhesion. This



is expected since, at these conditions, the surface coverage is negligible ( $\ll 1$  percent) for a  $\Delta H$  of 12 kcal/mole as given (ref. 16) for copper and carbon monoxide. At  $P_{CO}$  of  $10^{-1}$  torr at  $25^{\circ}C$ , partial coverage (about 20 percent) should occur and probably accounts for the loss of adhesion observed. However, for this latter data, the impurity and testing effects previously discussed should not be discounted and may also be partly responsible for the decrease. From equation (5), decreasing the surface temperature from  $25^{\circ}C$  to  $-120^{\circ}C$  increases the residence time of carbon monoxide on copper by about  $10^9$  (to residence times of years). Surface coverage will be governed by equation (7) and will be dependent on integrated exposure. The low-temperature experiment clearly shows the effect of exposure to carbon monoxide on adhesion of copper in that at slightly more than  $10^{-4}$  torr sec there is an abrupt decrease in adhesion that must be associated with increased adsorption of carbon monoxide. The leveling out of  $\alpha$  at  $P_{CO}$  of  $10^{-7}$  torr possibly indicates an approach to the equilibrium surface concentration corresponding to this temperature, while exposures to higher pressure ( $10^{-6}$  and  $10^{-5}$  torr) cause greater coverage and, thus, lowered adhesion. Generally, initial and subsequent values of  $\alpha$  under low temperature conditions were smaller than those observed at room temperature. The results in figure 6 (as well as fig. 7) were plotted in terms of  $\alpha$  rather than  $\bar{\alpha}$  because data were insufficient for correcting  $\alpha$  for mechanical effects at low temperatures. These results include some effects of mechanical testing.

In figure 7, results are presented of a typical experiment which demonstrates the near-reversible nature of carbon monoxide adsorption and desorption on copper as determined from the variations in adhesion under test conditions in which temperature and gas pressure are the primary variables. The data shown were collected in the sequence noted, from one specimen. Carbon monoxide pressures are indicated alongside the various segments of the data, while temperatures are indicated by open and closed symbols, the former corresponding nominally to  $-125^{\circ}C$ , the latter to room temperature. It is seen that when pressure is increased, adhesion is decreased (cycles 1 through 22). Adhesion may be partially regained either by lowering the pressure of carbon monoxide (cycle 23) or by heating the specimen (cycle 37). Data beginning with test 47 show the effect of cooling and then heating the specimen at a constant pressure of  $2 \times 10^{-5}$  torr. When the specimen was cooled to about  $-125^{\circ}C$ ,  $\alpha$  dropped to about 0.02 and when reheated recovered to about 80 percent of the test 47 adhesion. It is probable that the results observed arise from changes in the carbon monoxide coverage on the copper due to changes in pressure and temperature, as would be indicated by equations (5) and (6). These results are in accord with the observations of others on the nature of the adsorption of carbon monoxide by copper. For example, infrared and surface potential studies have indicated that the adsorption is "strongly physical" (refs. 21, 22) and is partially "stable irreversible" and partially weak reversible (ref. 23).

In a number of experiments similar to that shown by figure 6  $\alpha$  was monitored as a function of either temperature at constant pressure or pressure at constant temperature. It is seen (from substitution of eq. (5) in (6)) that, if the temperature and pressure conditions necessary to effect a loss of adhesion are known (assuming approximately monolayer coverage is sufficient to cause complete loss), the heat of adsorption of carbon monoxide on copper in

these experiments might be determined. The heat of adsorption as a function of temperature and pressure is given by

$$\Delta H = RT_s \ln \frac{\sigma(MT_g)^{1/2}}{3.5 \times 10^{22} sP\tau_0} \quad (9)$$

$\tau_0$  is  $2.0 \times 10^{-13}$  sec, as evaluated from the Lindermann relation (ref. 12) for carbon monoxide, and  $M$  is 28 g/mole. The value of  $\sigma$  was calculated to be  $1.9 \times 10^{15}$  molecules/cm<sup>2</sup>, assuming single site adsorption of carbon monoxide on copper. Corrections to the product  $sP$  discussed under the results on aluminum were assumed to apply here, namely  $sP = 0.02 P_{\text{measured}}$ . This correction increased  $\Delta H$  by  $\ln 50 = 3.9$ . The temperature of the carbon monoxide  $T_g$  is not known but is assumed to be midway between room temperature and the specimen temperature,  $T_s$ . Thus, equation (9) evaluated in terms of  $T$  and  $P$  is

$$\Delta H = 36.2 T_s \ln[(T_g)^{1/2}/P] \text{ cal/mole} \quad (10)$$

Heats of adsorption from six experiments at various temperatures and pressures, calculated from this equation, are tabulated in table II and have a mean value of 11.4 kcal/mole. The heat of adsorption of carbon monoxide on copper has been reported to be quite variable: 9.3 (ref. 13), 20 (ref. 24), and between 10 and 20 (ref. 25) (all in kcal/mole). The value determined in the present experiments is thus within the range of those previously reported. This suggests that the analysis and assumptions underlying the present determinations of  $\Delta H$  were reasonable and further clarifies the role of adsorbed gases in affecting the adhesion of metals.

Titanium, magnesium, and lead.— The adhesion of titanium, magnesium, and lead was studied in various environments and the results are shown in figures 8, 9, and 10. The adhesion behavior of these three metals differed in many respects from the results described above for aluminum and copper, but nevertheless, substantiated the general finding that adsorbed gases cause loss of adhesion. Only the more important findings and the results which differ from those for aluminum and copper will be discussed.

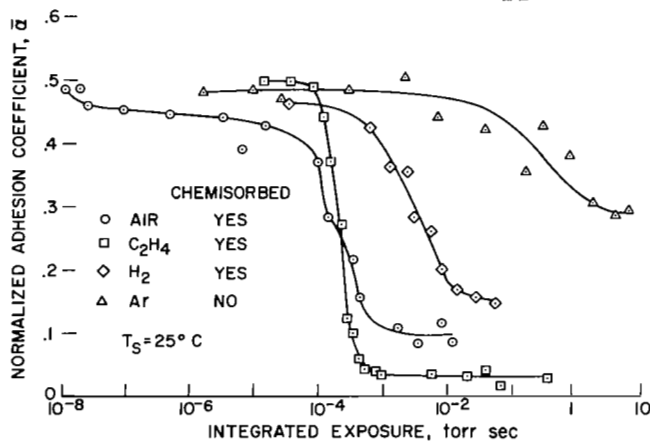


Figure 8.— Effect of various gas environments on the normalized adhesion coefficient of 99+ percent titanium.

Titanium, like other hexagonal metals (ref. 26), has, relative to cubic metals, an initially small  $\bar{\alpha}$  (table I) which decreases considerably with repeated adhesion testing in ultrahigh vacuum. Nevertheless, plotting  $\bar{\alpha}$  against integrated exposure (fig. 8) shows the effect on adhesion of the three chemisorbable gases (ref. 16) used (air, hydrogen, and ethylene). As expected, argon had noticeably less effect on adhesion than the three chemisorbable gases. The loss of adhesion under long exposures in argon was again attributed to impurities in the gas.

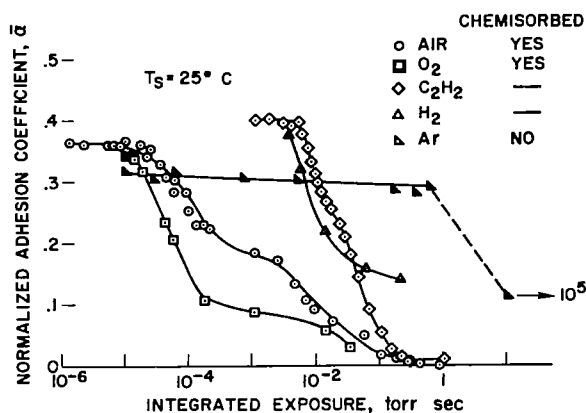


Figure 9.- Effect of various gas environments on the normalized adhesion coefficient of 99.95 percent magnesium.

Magnesium, another hexagonal metal displaying a small  $\bar{\alpha}$ , also showed varying behavior in different gas environments (fig. 9). Air, oxygen, and ethylene environments clearly affected, although at different integrated exposures, the adhesion of magnesium, while argon showed little effect. The data further indicate, but not conclusively because of the limited test, that the effect of hydrogen on adhesion is similar to that of ethylene and thus, apparently is not as readily adsorbed as oxygen. Except for oxygen (which is known to be chemisorbed, ref. 16), and argon (which is not), information was not found in the literature on the surface interaction between these various gases and magnesium.

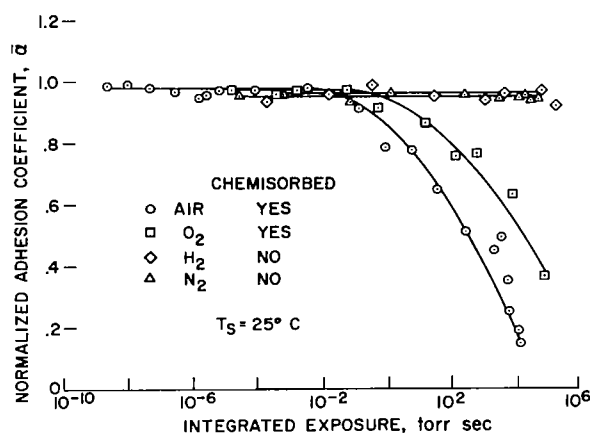


Figure 10.- Effect of various environments on the normalized adhesion coefficient of 99.99 percent lead.

Lead differed from other metals in that it displayed (fig. 10) an  $\bar{\alpha}$  of nearly unity (which, by the way, was virtually unaffected by mechanical testing effects - table I) and was insensitive to environment at exposures less than  $10^{-2}$  torr sec. As expected,  $\bar{\alpha}$  was particularly stable in nitrogen and hydrogen which have low heats of adsorption (ref. 14). Lead did not lose adhesion in air or oxygen until after exposures of about  $10^{-2}$  torr sec, considerably greater than the  $10^{-4}$  to  $10^{-3}$  torr sec exposures seen with other metals. Although no adhesion was found between lead surfaces fractured in air at 1 atmosphere some hours before testing, lead specimens fractured and promptly tested in air exhibited measurable adhesion. These results are not

due to lack of adsorption of oxygen, since it is known to be rapidly chemisorbed (ref. 13). Rather, it appears that this insensitivity might be due to the fact that a relatively hard oxide forms over a soft substrate. The possibility of cracking and subsequent burial of this oxide through specimen contact exists, which could result in fresh, clean surface being exposed, until a good deal of oxidation has occurred. With lead, this welding in air has been seen in friction and other adhesion studies (refs. 3, 27).

Gold. - Gold, under ambient conditions, does not chemisorb oxygen (refs. 16, 28, 29). Thus, gold was expected to show an adhesion behavior in oxygen that was distinctly different from other metals tested. Principal

results from tests (two specimens) on gold surfaces formed by fracture in UHV were: (1) Under UHV conditions, gold behaved much the same as copper, as shown in table I, (2) an oxygen environment (with apparent impurities) had little effect on adhesion to about  $10^{-2}$  torr sec exposure, beyond which a significant decrease occurred, and (3) gold exhibited no change in  $\alpha$  up to at least 1 torr sec exposure to hydrogen or nitrogen, gases which were not expected (ref. 14) to chemisorb at room temperature. Although the oxygen environment had less effect on the adhesion of gold than on the other metals examined, it nevertheless produced a greater effect than expected. The loss of adhesion above  $10^{-2}$  torr sec exposure was attributed to the formation of carbon monoxide from a reacting ion-gage-filament with oxygen (carbon monoxide is chemisorbed by gold (ref. 13)).

In other adhesion experiments the surfaces of gold disks were brought into contact under various environmental conditions. The results of these tests are summarized in table III. To explain these results, it is necessary to postulate the following: Since virtually no adhesion was found for the machined, chemically cleaned (in dilute nitric acid, then ethanol) surfaces when tested directly in laboratory air, the surfaces were evidently contaminated. Normal air contains many minor constituents, such as water, carbon dioxide, carbon monoxide, sulfur dioxide, and hydrocarbons, all of which gold chemisorbs; in addition, some grease and other residue can be expected to remain on the surface after the cleaning process. It is evident that high vacuum alone (see condition 2) is not sufficient to remove these contaminants, and that repeated contact of the specimens will produce a small amount of weldable surface while heating to  $300^{\circ}$  C is somewhat effective in removing these impurities. Impulsive loading in the normal direction, as expected (ref. 26), caused a large increase in adhesion probably by impurity burial. Significant adhesion was noted in an "air" (dry, hydrocarbon free) environment at relatively high pressure when the environment lacked chemisorbable species and was composed exclusively of oxygen, nitrogen, and noble gases.

#### CONCLUDING REMARKS

The effect of gas environment on the adhesion of a given metal is a specific phenomenon that depends mainly upon the nature of the particular gas-metal interaction. This was demonstrated by the adhesion of initially clean surfaces in environments of gases having various heats of adsorption and causing various degrees of contamination, under differing test parameters (temperature, pressure, and exposure). Adhesion in those metal-gas systems having heats of adsorption above 20 kcal/mole was dependent (with the exception of lead-oxygen) on integrated exposure (pressure  $\times$  time). Loss of adhesion in these environments occurred at exposures of  $10^{-4}$  to  $10^{-2}$  torr sec. Effects on adhesion in physically adsorbed gases (heats of adsorption of around 5 kcal/mole or less) were noted only at much higher exposures and then, quite probably, were due to adsorption of impurities in the gases, or to mechanical changes in the specimen. Adhesion in metal-gas systems with heats of adsorption between 5 and 20 kcal/mole could be lost at low temperatures and/or high gas pressures, again conditions causing increased surface coverage. Two metals showed somewhat unusual behavior: Lead, because of its particular

mechanical properties, exhibited exceptionally high adhesion in various adsorbable gas environments, and gold, because it does not chemisorb oxygen under normal conditions, can if cleaned of other adsorbed species, self-weld in dry air.

The results of this study provide some guidance in selecting materials to avoid adhesion. It is evident that since the adhesion of a metal depends on its gas environment, the successful operation of contacting metal components depends on interaction of the environment with the metal. Since the adhesion of metals (under a given set of pressure, exposure, and temperature conditions) in gases with large heats of adsorption is considerably less than in gases with small heats, fewer problems traceable to adhesion would be expected from a metal part operating in the former type of environment. The relatively trouble-free operation holds not only for "active" gas environments (such as oxygen or air) but also for oils and greases, which perform much the same function as adsorbed gases by keeping metal atoms from contact across the surface interface. Conversely, difficulties would be expected from parts operating in gas environments which are not chemically adsorbed by the metal. Thus, from the present results, problems due to adhesion might be expected from metal-gas environment combinations such as gold-oxygen, copper-hydrogen, aluminum-nitrogen, and, of course, from all metals which must operate in ultra-high vacuum. Since the minimum exposure for formation of a "protective" surface layer in an active gas is about  $10^{-4}$  torr sec, an exposure must exceed this before clean surfaces can be contacted without some adhesion resulting. For many metal-gas combinations, minimum exposure necessary to avoid adhesion is much longer than  $10^{-4}$  torr sec and, in numerous cases, adhesion occurs only under unusually high-pressure or low-temperature conditions. Although the present results do provide some guidelines for material selection, it is apparent that they must be used in combination with results of other types of adhesion studies, that is, studies which provide data on the removal or protective nature of surface layers (such as oxides, nitrides, or lubricants) under various environmental conditions.

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TABLE I.- EFFECT OF MECHANICAL TESTING IN UHV ON THE ADHESION COEFFICIENT  
AT ROOM TEMPERATURE OF THE METALS INVESTIGATED IN THE PRESENT STUDY

<u>Metal</u>	<u>Adhesion Coefficient</u>	
	<u>Initial</u>	<u>After 50 cycles</u>
Aluminum	0.84	0.6
Copper	0.76	0.5
Gold	0.78	0.5
Lead	0.98	0.95
Magnesium	0.35	0.15
Titanium	0.48	0.2

TABLE II.- THE HEAT OF ADSORPTION OF CARBON MONOXIDE ON COPPER AS  
DETERMINED FROM TEMPERATURE AND PRESSURE VALUES OF CARBON  
MONOXIDE NECESSARY TO CAUSE LOSS OF ADHESION IN COPPER

<u>Sample</u>	<u>Pressure, torr</u>	<u>Temperature, °K</u>	<u>Heat of adsorption, kcal/mole</u>
1	$2.5 \times 10^{-1}$	248	11.0
2	$6 \times 10^{-4}$	212	12.0
3	$1 \times 10^{-5}$	176	11.3
4	$1 \times 10^{-5}$	160	10.2
5	$1 \times 10^{-6}$	183	12.6
6	$1.1 \times 10^{-7}$	155	11.4
Mean			$11.4 \pm 0.8$

TABLE III.- THE ADHESION OF GOLD UNDER VARIOUS TEST CONDITIONS

<u>Condition</u>	<u>Adhesion coefficient</u>
1. Initial contact in laboratory air	$\ll 0.001$
2. Initial contact in UHV after 10 hrs at $10^{-9}$ torr	$\ll 0.001$
3. After 50 contacts in laboratory air	$< 0.001$ (maximum)
4. After 50 contacts in UHV	0.002 to 0.005
5. Contact at 300° C in UHV	0.08
6. Contact after cooling to 25° C following (5) in UHV	0.02
7. Contact after impulsive loading at 300° C in UHV after (6)	0.5
8. Contact after cooling to 25° C following (7) in UHV	0.25
9. Contact following (8) after 100 torr "air" for $10^3$ sec	0.1